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MONTHLY PROGRESS REPORT

No. 3

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Submitted by

NEW PRODUCT RESEARCH of TAPCO

OTS PRICE

XEROX	\$ <u>1.10</u> <i>ph</i>
MICROFILM	\$ <u>0.80</u> <i>mf</i>

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I. INTRODUCTION

This document represents a third monthly report covering an experimental program for development of an "Osmotic Still" and improvement in the performance characteristics of the Ionics Dual Membrane Fuel Cell and the work completed during the month of September, 1962. This development work is being accomplished under NASA-Lewis Research Center Contract No. NAS 3-2551 by the New Product Research Department of Tapco and Ionics, Inc. as a subcontractor to Tapco.

II. OVERALL PROGRESS

1. Test rig assembly needed for development tests of the "Osmotic Still" has been completed and checked out. Schematic diagram of test rig is shown in Figure I of Monthly Progress Report No. 1 published in August.
2. Materials used in the "Osmotic Still" test unit and acid pump construction such as Polyvinyl Dichloride and Cast Methyl Methacrylate have undergone precautionary compatibility tests to determine any possible adverse reaction to acid. Results of these tests are presented in Section V of this report.
3. The design of an improved single fuel cell unit has been completed. It has been possible to incorporate a number of design features which will have direct applicability to the multiple cell configuration to be constructed in another phase of the project.
4. A materials testing program for development of fuel cells is underway. Duplicate samples of platinized titanium, titanium, Tri-Lox mesh and epoxy gasketing material have been immersed in $6N\ H_2SO_4$ and the resulting assemblies maintained at 30° , 60° and $95^\circ\ C$. All samples were weighed before immersion and will be weighed after removal from bath in order to ascertain quantitatively the effect of degradative action. In addition, visual observation in the form of microscopic analysis will be performed.
5. Purchase of material and dies needed for single cell construction is almost complete.
6. The construction of the control and regulatory systems for fuel cell development is underway.
7. The engineering survey on the strength and compatibility of various structural and operation components in $6N\ H_2SO_4$ is continuing.
8. Utilizing some 2" x 2" test cells, preliminary data have been obtained on the durability of the "sintered" electrode discussed in the report of September 10, 1962. Although performance drops off somewhat, the total cell voltage at given current densities continues to be higher than that usually obtained from similar 2" x 2" cells with pasted electrodes. The performance of one of our "sintered" electrode cells is shown in Figure 1. It is interesting to note that after the cell "rests", i.e., when hydrogen and oxygen fuel supplies are shut off and then resumed, the device returns to its original voltage output. This reveals that the change in operating characteristics is reversible and is probably due to the slow migration of H_2SO_4 into the membrane, particularly in the area of the oxygen electrode where water is being formed. Two approaches are underway to eliminate the degradation in performance:

II. OVERALL PROGRESS (Cont'd)

8.
 - a. Increased temperatures (35-40°C) have been utilized in order to avoid what appears to be a diffusion controlled problem. Preliminary results shown in Figure 2 appear to make this approach promising.
 - b. The molecular network in the membrane will be increased in diameter to allow more rapid diffusion of H_2SO_4 .

III. CURRENT PROBLEMS

1. It was found when checking out "Osmotic Still" testing that after subjecting the Trilox (U.S. Rubber Co.) spacer fabric (Polyethelene and Saran), which is located between the membrane and the membrane perforated support disk (see Figure 2 of Monthly Progress Report No. 1), to 200°F temperature and about 15 psi pressure for 6 hours and room temperature and 15 psi pressure for 18 hours, the fabric thickness was decreased and assumed a permanent deformation. The thickness was decreased from 0.1 inch to 0.075 inch. This decrease in spacer thickness resulted in stretching of the simulated membrane. The stress of this magnitude when imposed upon the working membrane will result in working membrane failure. As a result, it is envisioned that changes in housing component design and exploratory type tests with actual membranes will have to be run to eliminate this membrane stress before actual water removal tests can proceed.
2. The probable attack by 6N H_2SO_4 on platinized titanium used in fuel cells at higher temperatures (95°C).
3. The deterioration of gasketing plastic at 95°C in fuel cells.
4. Deterioration of cell performance due to lag in diffusion of H_2SO_4 into region of water formation at oxygen electrode.

IV. NEXT MONTH'S EFFORT

1. "Osmotic Still" tests will be conducted with Ionics' Cation working membranes.
2. Placing on test a minimum of two single cells to serve as controls for further work.
3. Completion of 3 fuel cell test rigs with accompanying controls and regulating devices.
4. Continued materials analysis and testing necessary for development of fuel cells.

V. TEST RESULTS

1. "Osmotic Still" test rig was checked out in the following manner. The epoxy impregnated glass fiber sheets were inserted into the still instead of working membranes in order to eliminate a possible source of leakage in the still itself. If any leaks occurred after insertion of epoxy reinforced glass sheet, it would indicate that there is a leak somewhere in the test rig system.

The first checkout of the system was with water instead of electrolyte. Water was circulated at 200°F and 75 centimeters Hg pressure gage in order to check out electrolyte pump operation at high temperature and air leakage through the

V. TEST RESULTS (Cont'd)

1. pump. At the same time, a hard vacuum of 74 centimeters of Hg was pulled on the vapor side of the "Osmotic Still" housing and the system was closed to the atmosphere. Under these conditions of operation, it was necessary to increase the tolerance in the pump shaft to keep it from seizing. A number of pressure and vacuum leaks developed during subsequent tests which were located and corrected. The final check of the system consisted of pulling a vacuum of 74 centimeters of Hg, and closing off the system to the atmosphere. After 16 hours of soaking at the above condition, the vacuum in the system decreased to 67 centimeters Hg, which was considered as adequate in this development work.
2. Precautionary compatibility tests consisted of immersing two samples of material in the same 60% sulphuric acid bath for a continuous 31 day test. One sample of Polyvinyl Dichloride (2.905 grams) and one sample of Cast Methyl Methacrylate (2.462 grams) were used for testing purposes. During the period of 31 days the acid bath was heated to a temperature of 200° F and was kept at that temperature during the working hours for a total of 143 hours and at room temperature for the remainder of the time. Visual inspection after this elapsed time showed no deterioration of samples. It was intended to weigh these samples at the end of test but the samples were accidentally destroyed due to overheating of the acid bath.
3. A number of experiments using "sintered electrodes were performed as discussed in Section II-8. The "sintered" electrodes were prepared as described in the September 10, 1962, report, page 4. One cell was placed on life test and the voltage and amperage recorded as a function of time. Table I contains data obtained at 16 ma/cm² and Figure I is a graphical representation of these data.
4. In another experiment a cell with "sintered" electrodes was immersed in an oil bath at temperatures ranging from 24°C to 44°C. The voltages recorded in this experiment at 16 ma/cm² are shown in Table II. Figure 2 is a graph of these data.

VI. QUALITY ASSURANCE

Test Stand Build has been completed and is ready for operation. Vacuum test for leakage in the stand system presented a temporary problem but has been satisfactorily corrected. RD&E meter lab technicians calibrated the thermometers. The readings have been recorded and entered into the schedule for a periodic recheck to assure constant accuracy.

As mentioned in the Quality Assurance Report for September, 1962, the surveillance trip to Ionics, Inc. at Cambridge, Mass., was accomplished on September 11 and 12 in the company of NASA representatives. Copy of the trip report is attached as part of this report.

A follow-up trip to Ionics by a TRW Quality Control representative is planned for the week of November 12, 1962.

TABLE I

LIFE TEST OF 2" x 2" CELL 9204 - SINTERED ELECTRODESTemperature 24-26°C

<u>Hours of Operation</u>	<u>Open Circuit Voltage</u>	<u>Load Voltage</u> <u>16 ma/cm²</u>
0	--	0.78
24	1.04	0.83
48	1.07	--
312	1.03	0.77
408	1.02	0.72
480	1.02	0.80*
648	1.05	0.715
864	--	0.68

*Cell operation was stopped during week-end when oxygen supply was exhausted. This reading was taken shortly after operation was resumed.

TABLE II

TIME-TEMPERATURE STUDY ON 2" x 2" CELL 9209 - SINTERED ELECTRODES

Current Density - 16 ma/cm²

<u>Temperature</u>	<u>Voltage</u>
24°C	0.76 V
35	0.797
36.3	0.80
39.5	0.805
41.5	0.810
44.0	0.818

OPERATING CURVE OF 4-INCH² FUEL CELL (Voltage vs. Time)

Cell 9204 - Sintered Electrodes

Temperature - 24-26°C

Current Density - 14.9 amps/ft²

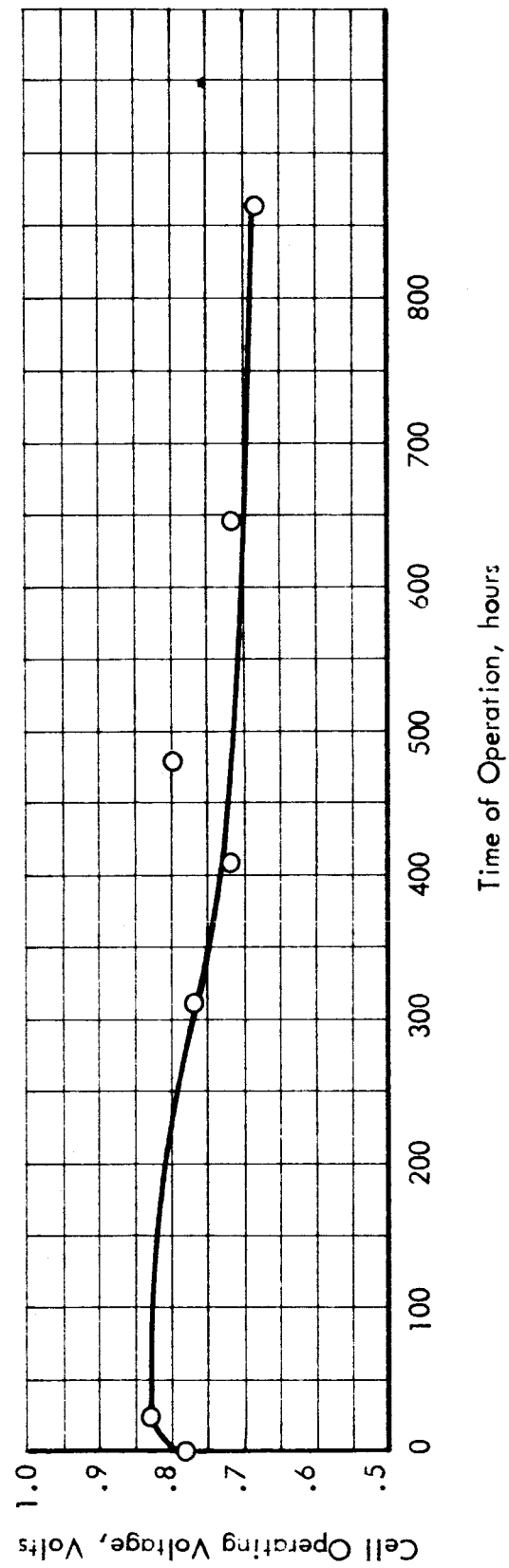


Figure 1

EFFECT OF ELECTROLYTE TEMPERATURE ON CELL VOLTAGE

Cell 9209 - Sintered Electrodes
Current Density - 14.9 amps/ft²

